THE EFFECT OF LOW FREQUENCY CYCLING & MILL SCALE ON STRESS CORROSION CRACKING OF PIPELINE STEEL IN SIMULATED FUEL GRADE ETHANOL

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THE EFFECT OF LOW FREQUENCY CYCLING & MILL SCALE ON STRESS CORROSION CRACKING OF PIPELINE STEEL IN SIMULATED FUEL GRADE ETHANOL

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SCC	Stress Corrosion Cracking
FGE	Fuel Grade Ethanol

ABSTRACT

Using non-renewable fossil fuel energy resources has become a major concern in modern day society. Major efforts have been made to decrease the effect of such hazardous materials. Ethanol (CH^3CH^2OH, or EtOH) has been proven to be a promising alternative option to fuel. Approximately 10-15% of the commercial fuel used nowadays is composed of fuel-grade ethanol (FGE). However, field failures due to stress corrosion cracking (SCC) of carbon steel pipelines and storage tanks used in FGE transportation have been reported. Leaks are found at stress concentration points, such as heat affected zones and geometric discontinuities. Prior research on the effect of EtOH chemistry and electrochemical conditions on crack initiation, growth and propagation behavior has shown that contaminants and/or additives are important factors in causing SCC in FGE pipelines. The role of low frequency stress fluctuations on SCC initiation and propagation on the inner surface in FGE pipelines was not understood. The main objective of this research is to evaluate low frequency cyclic effects on SCC behavior under simulated conditions, and thus use the obtained information to optimize productivity and prevent catastrophe. A four-point bend test on a pipeline section immersed in an ethanol solution will be used to simulate the service conditions experienced during operation. It has been conjectured that SCC is not experienced with a static applied load below or above yield stress levels, therefore indicating that dynamic or cycling loading is required for SCC to occur. A smoother surface finish results in significantly less SCC than a rougher surface, emphasizing the importance of surface roughness in SCC behavior. A smaller R-ratio results in lower crack density, nucleation rate and crack velocity than larger R-ratios, hence indicating the importance of fluctuating stresses in SCC behavior. A higher cyclic frequency results in increasing crack density, nucleation rate, velocity and crack length, with a possible threshold leading to crack propagation. Longer test durations resulted in reduced crack velocity, indicating that cracks grow slowly with time due to a number of factors, including crack shielding. Finally, oxygen supply is essential for SCC to occur, which support previously conducted research by X. Lou *et. al* and Sridhar *et. al*.

CHAPTER 1
INTRODUCTION

Using non-renewable energy resources has become a major concern in modern day society. They pollute the water we drink, the air we breathe, and thus subsequently affect all aspects of our lives. Consequently, major efforts have been made to decrease the effect such hazardous materials. Ethanol (CH_3CH_2OH, or simply EtOH) has been proven to be a very promising additive/alternative option to fuel. Approximately 10-15% of the commercial fuel used nowadays is composed of ethanol. However, field failures due to stress corrosion cracking (SCC) of carbon steel pipes and tanks used in FGE transportation/processing have been reported. Failure points are mainly at stress concentrations (i.e. weld heat affected zones), where leaks are imminent. Such phenomena can lead to catastrophic outcomes. None of the failures were observed in the producer plants, but rather in the user facilities. Prior research on the effect of EtOH chemistry and electrochemical conditions on crack initiation, growth and propagation behavior has shown that contaminants and/or additives are important factors in causing SCC in FGE pipelines [1 to 10]. The role of low frequency stress fluctuations on SCC initiation and propagation on the inner surface in FGE pipelines was not understood.

The main objective of this research project is to evaluate cyclic and mill-scale effects on crack behavior under simulated conditions, and thus use the obtained information to prevent unexpected failures and predict lifetime cycles of existing pipelines, therefore optimize productivity and prevent catastrophe.

This thesis paper will begin by discussing the main mechanism behind pipeline failure and the physics behind it: stress corrosion cracking, or SCC. It will then discuss the procedure followed in order to simulate the pipeline conditions and thus provide an appropriate environment that can be used to analyze the crack behavior of the system. Afterwards, previous research findings will be discussed in more detail to facilitate subsequent discussions. Finally, the cyclic and mill effects are addressed in detail to further investigate crack behavior.

CHAPTER 2

LITERATURE REVIEW

Corrosion issues, especially stress corrosion cracking (SCC), have been an important problem in the pipeline industry for many decades. In 1978, Vosikovsky *et. al* analyzed the effects of corrosion fatigue on crack behavior in a crude oil pipeline [16]. This study was developed as a result of 4 field failures during a six month period, all of which originated from a pre-existing crack found in the inside toe of a longitudinal weld. Metallographic sectioning and nondestructive evaluation methods (i.e. scanning electron microscope) were used to analyze the failure surfaces. It has been conjectured that the weld areas were subjected to extensive plastic deformation during the pipe's mechanical cold expansion. This resulted in the growth of the pre-existing flaws into fatigue cracks, which propagated to critical sizes under service loading conditions, inducing the failures observed on the field. Defects, large and small, require many full pressure cycles to failure, assuming that the pipeline has a long life and a high shutdown frequency. However, shutdown occurrences result in overpressures, significantly shortening the life expectancy of the pipeline. Pressure fluctuations and a high frequency of shutdowns result shorten pipeline life expectancy even further, and is possibly one of the main reasons behind the failures experienced in these 4 different incidents. Hence, the presence of pressure fluctuations and high shutdown frequency, with the aid of pre-existing defects, result in significantly shortened pipeline lifetimes and consequential failures due to corrosion fatigue.

Nowadays, the incorporation of ethanol in the fuel blends is an ongoing effort to utilize alternative fuels along with conventional fuels to help reduce the environmental impact. Fuels used today have as much as 15% ethanol within the blend. Since pipelines are used to transport large amounts of fuel over distances that can range from a few miles across towns to thousands of miles across continents, the effect of the fuel's chemical constituents is essential to the understanding of SCC crack initiation and propagation behavior. X. Lou *et. al* analyzed the effect of water content, chloride content and oxygen content on SCC of X65 carbon steel in simulated fuel-grade ethanol [10]. Slow strain rate testing (SSRT) was used to evaluate such effects. It has been found that chloride content greatly affects SCC crack growth as well as crack initiation. Higher chloride concentrations led to increased crack velocities and crack densities. A chloride content ranging between 10^{-4}M and 10^{-3}M is required to render the specimen environment aggressive enough for SCC occurrence [18]. It has also been observed that the presence of oxygen plays a vital role in SCC initiation. The absence of oxygen resulted in the elimination of SCC altogether. Such observations are supported by the work of Sridhar *et. al*'s [15]. X. Lou *et. al* also found that water content affects the surface passivation of the steel specimens, with a transition from SCC to pitting when the water content weight percentage exceeded 2.5% [10]. Ethanol acidity (pHe) was also found to have significant effects on SCC behavior, with a more alkaline solution resulting in SCC initiation [10], while a more acidic solution, combined with higher chloride content, resulted in enhanced pitting growth [8]. In a separate study, X. Lou *et. al* also demonstrated that pitting is reduced when the water content exceeds 10 vol% [8]. Cyclic potentiodynamic polarization and open circuit potential monitoring were used to monitor the effects on the samples. As the water content increases, the formation of an iron hydroxide passivation film forms on the carbon steel surface. Conversely, with lower water content, iron oxide becomes a significant corrosion product on the sample surface.

In a different study, X. Lou *et. al* analyzed the effect of oxygen, hydrogen, acetic acid and water content on SCC of carbon steel pipelines [6]. It has been demonstrated that the main cathodic reaction involved in simulated fuel-grade ethanol (SFGE) for carbon steel is oxygen reduction. Hydrogen activity increases when de-aeration is dominant and at much higher cathodic over-potential than for oxygen activity. Hydrogen evolution is due to the presence of water, ethanol and acetic acid and high over-potentials during de-aeration. Increased water content increases proton dissociation rates, which in consequence increases the cathodic current density. Such conditions are hard to achieve under normal operating conditions. Water and acetic acid enhance cathodic reaction kinetics in the fuel-grade ethanol blend.

Sridhar *et. al* analyzed the effects of various chemicals on SCC behavior [1]. It has been conjectured that ethanol absorbs extensive amounts of water and oxygen if exposed to direct ambient air, even when exposed for short time durations (i.e. water reached 1%

wt after just 1hr). Contaminants such as acetic acid, chloride and methanol showed significant effects on the SCC of the steel specimens analyzed. However, the most significant of all contaminant is oxygen, which increased the corrosion potential drastically. Furthermore, galvanic contact raises the corrosion potential, which in consequence induces secondary redox reactions that enhance SCC. Nevertheless, galvanic contact alone is not sufficient to induce SCC altogether.

Sridhar *et. al* analyzed the effects of metallurgical and environmental parameters on SCC behavior in carbon steel in fuel-grade ethanol (FGE) [17]. It has been found that metallurgical parameters such as steel grade, heat affected zones (HAZ's), weld points…etc… do not have an important effect on SCC behavior altogether. It has also been found that SCC does not occur if the ethanol content is below 15% in an ethanol-gasoline blend. A 50% volume ethanol-gasoline blend has been shown to have higher SCC occurrence probability and crack growth rate than that in higher or lower ethanol concentration blends. Since oxygen is a major factor of SCC, various methods have been developed to dissolve oxygen within the ethanol-gasoline blend, such as vacuum degassing, nitrogen purging and the introduction of chemical de-aeration agents (i.e. oxygen scavengers) or electrochemical de-aeration agents (i.e. fine iron). Sridhar *et. al* also found that water content over 4.5 vol% prevents SCC, which further supports previous research papers discussed in this literature review.

Electrochemical behavior also plays an important role in the understanding of SCC behavior of carbon steel pipelines. X. Lou *et. al* analyzed the effect of phase angle on stress corrosion cracking (SCC) behavior of carbon steel in fuel grade ethanol (FGE) [7]. Slow strain rate tests as well as electrochemical impedance spectroscopy (EIS) were used to conduct and analyze crack behavior as well as electrochemical reaction mechanisms. It has been observed that phase angle at a low frequency (below 1 Hz) is more sensitive to SCC. A phase angle decrease is observed in regions with active crack propagation. The sample surface, crack wall and crack tip were analyzed in detail by following a transmission line model to simulate the EIS response for stress corrosion cracking using the geometrical features of the samples. Goodman *et. al* analyzed the effects of water content, chloride content, oxygen level and pHe on repassivation kinetics of X65 carbon steels in SFGE [14]. Scratch tests were conducted as the main approach for this work, using test coupons machined from X65 pipeline sections and fuel-grade ethanol following the ASTM D4806 protocol. It has been found that increased chloride concentration resulted in increased current density and decreased current decay rate in both scratched and unscratched samples. Repassivation is slower in SFGE than it is in other aqueous solutions. Oxygen removal resulted in extensive increases in current density, therefore making repassivation more difficult. Lower pHe resulted in no repassivation or decay after scratching is conducted. Preferential localized and pitting corrosion occurred in scratched regions where the passivation film was absent.

Mechanical effects are also important in the understanding of carbon steel SCC behavior. X. Lou *et. al* demonstrated that slower strain rates in mechanical cyclic loading resulted in increased crack density and crack lengths and decreased crack velocities [10]. Metallurgical inclusions found in X65 carbon steel, such as silicate and alumina, were found to serve as crack initiation sites due to the fact that higher plastic deformation is experienced near such inclusions [10].

Pipelines undergo pressure fluctuations during operation, which result in cyclic loading that is detrimental to the lifetime of the pipeline [16]. Thus, cyclic effects play a major role in SCC initiation and propagation. Sowards *et. al* analyzed the effect of cyclic loading carbon steel pipelines in simulated fuel-grade ethanol (FGE) [13]. The analysis is done using two pipeline steel types (X52 and X70) as well as a storage tank steel (A36). Fracture mechanics was used to develop a feasible model to predict the life expectancy as well as the structural wellbeing of the system. Conventional tensile testing was conducted (following ASTM E8/E8M) on compact tension (CT) specimens, which were machined according to ASTM E647. The specimens were pre-cracked and the crack propagation rates were monitored and calculated accordingly (following ASTM E647). Cyclic parameters such as the R-ratio and frequency were 0.1 and 10Hz (in air) or 0.1Hz (in FGE), respectively. Results have shown that fatigue crack growth rates increased with increasing stress intensity levels (i.e. as the crack in the specimen grows, presenting a bigger stress concentration at the crack tip) in both air and FGE in all three steel types, with FGE following a more nonlinear pattern than does air. The cracks were typically of trans-granular nature. It has also been conjectured that crack growth rates decrease with increasing loading frequency (from 0.01Hz to 0.1Hz).

The frequencies analyzed by Sowards *et. al* represent values that are much higher than those experienced in real-life situations. Hence, the effect of low frequency (that is more realistic) is not yet understood, and no prevalent research has been conducted on such a topic. Hence, the purpose of this study is to analyze the effects of low, real-life frequencies on SCC behavior. Such a study will aid in a better understanding of how real-life cyclic conditions affect SCC crack initiation and propagation. As a result, a better prediction of the lifetime of the pipelines can be obtained, therefore aiding in preventing unwanted and unpredictable failures to occur.

CHAPTER 3
THE MECHANISM

Stress corrosion cracking is a corrosion phenomenon, which induces brittle mechanical failure at low tensile stresses (much lower than the material's yield strength) of an alloy exposed to a corrosive environment. Three conditions are required for SCC to appear: a corrosive environment, a susceptible material and present tensile stresses. Depending on these three conditions, SCC can be mild or fairly severe, resulting in crack formation after very few stress cycles.

In this application, a pipeline network made of X-65 and X-66 carbon steel is used to transport Ethanol (CH_3CH_2OH, or simply EtOH) throughout the United States of America. Over 1 million miles of pipelines are used in this multi-billion dollar industry. Figure 1 illustrates the US pipeline industry map.

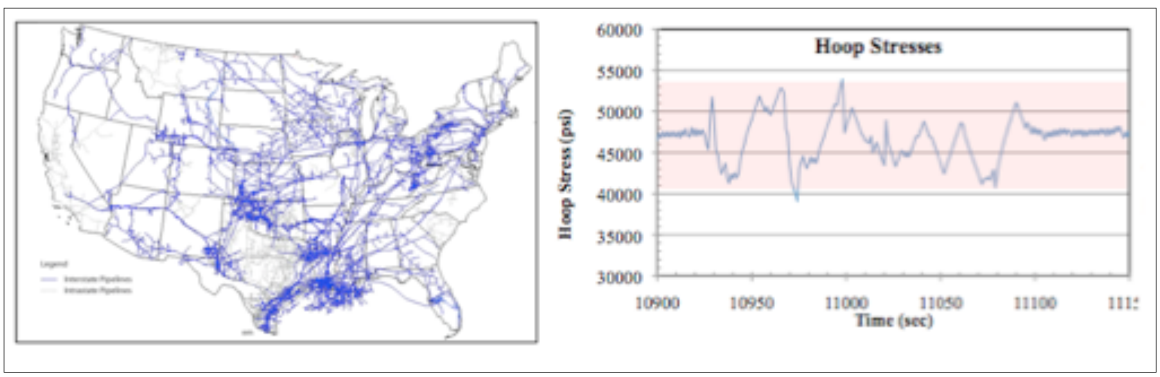

Figure 1. Pipeline Transportation Map of the United States

The ethanol is pumped through the pipeline network using a multitude of pumping stations located miles apart. From our fluid mechanics knowledge, it is known that pressure losses are experienced from one station to the next as the fluid is pumped through. From our mechanical behavior of materials knowledge, it is known that cylindrical systems experience hoop stresses exerted by the passing fluids. According to data obtained from pipeline operators, pipelines experience low frequency hoop stress cycling. Such stress fluctuations result in an increase in the creep rate, a phenomenon also known as the Kennedy effect [11]. An example of such data is illustrated in Figure 1.

From the figure, it can be observed that the material experiences variable cyclic loading in some occasions during operation.

During operation, an unfortunate and unavoidable fact is that oxygen, chlorides and other contaminants are introduced into the system. The presence of oxidizers and volatiles will bring the electrochemical potential of the pipeline steel down to the active region. As a result, the material will not be able to form a passive film, which protects the material from further corrosion. This is analogous to rust forming on iron when left in the air for some time. The rust is not the material degrading, per say, but rather a thin layer of metal oxide, which protects the metal from further degradation. When in the active region, this passive layer is continuously destroyed, resulting in gradual and continuous material degradation.

As mentioned before, field failures have been mainly in regions where high stress concentrations were present. Examples include changes in pipeline geometry (turns, cross sectional changes…etc…), heat affected zones (HAZ's) resultant from welds, which introduce residual tensile stresses and impurities resultant from the welding process. This indicates that such spots are especially vulnerable and are significantly weaker than other sections of the pipeline network.

Due to internal flaws present in any and all materials (and especially metals such as carbon steel), cyclic loading conditions may result in crack initiation. For metals (i.e. X-65 carbon steel) under loading, such internal flaws (combined with dislocation motion and slip band formations) grow and coalesce to form the initial cracks. Cyclic loading makes crack formation much easier and much faster than constant load conditions.

Thus, it can be concluded from this section that the pipeline steel, which is X-65 carbon steel in this case, when subjected to cyclic hoop stresses and loadings in a corrosive ethanol environment (due to oxidizers and volatiles), may result in the formation and propagation of cracks, which ultimately lead to system failure. Such cracks form where structural integrity is most compromised (i.e. stress concentration areas).

CHAPTER 4
PROCEDURE / EXPERIMENTAL

A four-point bend test is used to simulate the stress conditions on the inner surface of a pipeline. This test is conducted in an ethanol solution to simulate the corrosive environment. An MTS servo-hydraulic load-frame equipped with a 10-kip actuator is used as the main stress source for this experiment. A sectioned piece of a pipeline test sample made of X-65 carbon steel is used as the component tested for SCC. The standard protocol followed for this experiment is ASTM G39-99. The corrosive solution is prepared by mixing 1L of 200-Proof EtOH with 0.067g of sodium chloride (NaCl). The salt is introduced in the solution in order to simulate the effect of chlorides and other contaminants that can and will enter the pipelines as the ethanol is transported. The 200-proof ethanol is selected due to its water content being in the range of 650-750ppm. Due to the fact that ethanol has a low polarity and NaCl being an ionic compound, the solution is stirred for at least 24 hours in order to ensure complete dissolution of the chlorides in the ethanol solution. Afterwards, a sample of the solution is extracted and stored for record and inventory purposes.

The stainless steel container, in which the test fixture and the sample is installed, is washed and rinsed thoroughly with water and soap and extensively sanitized with acetone to ensure any soap and water particles are not contained in the crevices of the container. Afterwards, the container is left to dry for at least 24 hours to ensure that no water molecules are still present.

The test sample is properly cleaned by installing it in an acetone solution contained in a beaker, which is then installed in a Sonicor SC-101th Sonicator for 10 minutes to ensure any dust or other foreign contaminants are cleaned off. The sample is then fixed onto the four-point test fixture inside the cleaned, dry container. The container is then filled with the ethanol solution and is then closed and locked securely. Afterwards, the container is installed into the load-frame testing area and is subjected to a simulated fatigue test. This test involves subjecting the sample to cyclic loading typically ranging from a maximum load of 6,500lbf to a minimum load of 200lbf. A constant air purge is introduced in order to account for the effect of constant oxygen supply in the pipeline.

After the test is complete, the sample is sectioned using a high-speed saw and polished in order to analyze the stress corrosion cracking susceptibility. A sample of the ethanol solution is extracted after the test is complete. This is purely for record and inventory purposes. The polishing process for post-test analysis of epoxy-mounted sections of the test sample involves successive grinding using a Buehler Ecomet 6 grinder. The grinding involves 120, 400, 600, 1000 and 2000 grit polishing sandpaper. Afterwards, the ground samples are installed in a Buehler Vibromet 2 for 30 minutes for finer polishing down to 0.05 microns surface roughness. After the polishing process is complete, the samples are sonicated in an acetone solution to ensure that no contamination occurs to the fracture surface. Finally, the polished samples are observed under the microscope to quantify the SCC crack behavior of the tested sample. Table 1 illustrates the metallurgical composition of X-65 carbon steel used in this study.

Table 1. X-65 Carbon Steel Metallurgical Composition [19]

Al	C	Fe	Mn	P	S	Si
0.041	0.08	98.367	1.26	0.01	0.002	0.24

Figure 1 illustrates the main apparatus used, while Figure 2 is a schematic of the test's nature (how it is conducted).

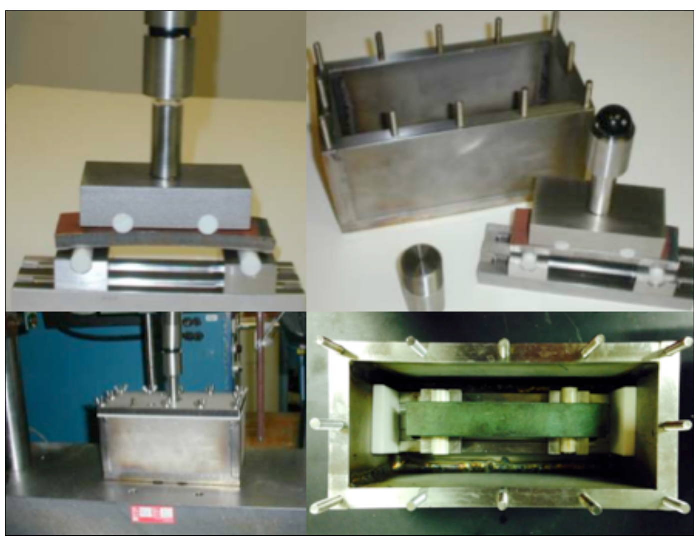

Figure 2. Main Apparatus

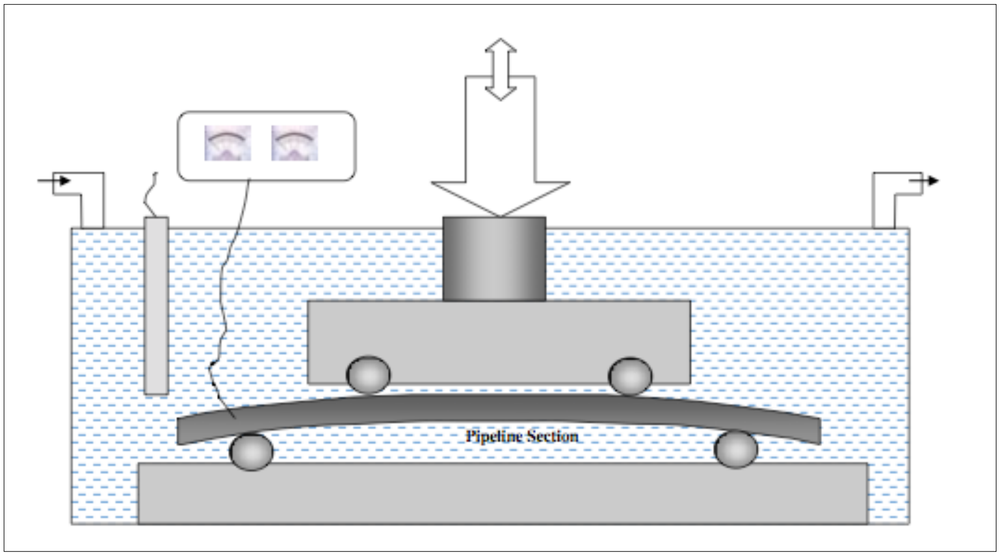

Figure 3. Four-Point Bend Test Schematic

Figure 3 illustrates before and after effects of the test as well as the sectioning and sample preparation/polishing for surface analysis. The surface to be analyzed is highlighted in red. Figure 4 illustrates the crack surface that is analyzed after conducting the test. The cracks can be clearly seen from the micrographs obtained.

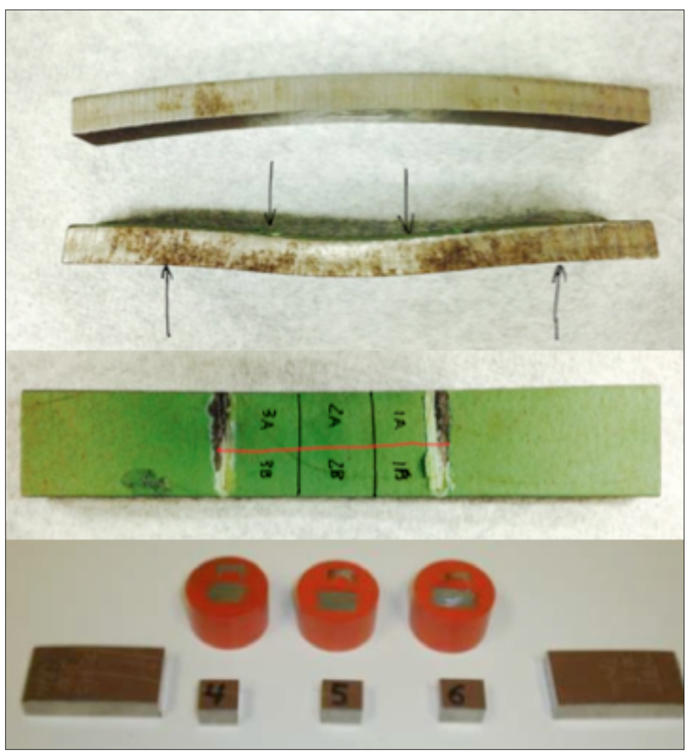

Figure 4. Sample Sectioning & Surface Preparation

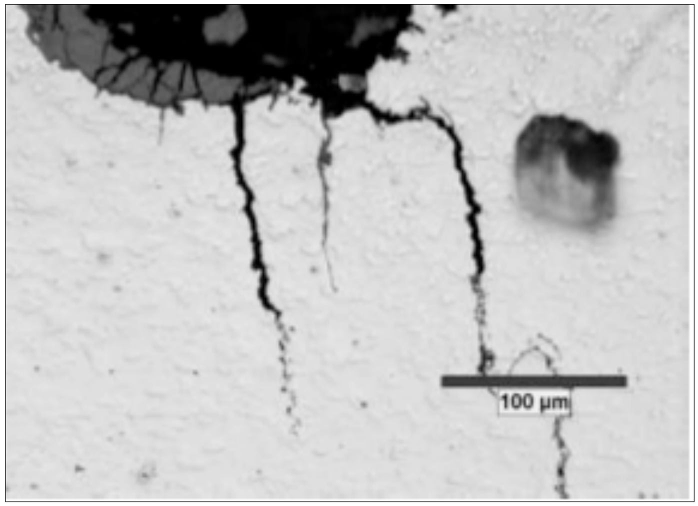

Figure 5. Analyzed Crack Surface

Figure 5 illustrates the load-displacement and load-time relationships of the sample during the course of the four-point bend test. It can be observed from the load-displacement test that the displacement keeps growing as more cycles are experienced, indicating that cracking/mechanical performance depreciation is occurring.

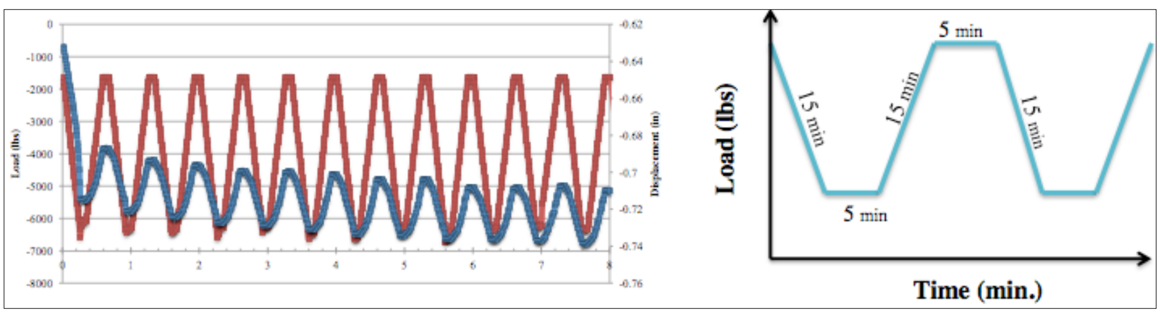

Figure 6. Load vs. Displacement & Load vs. Time Relationships

The crack surface is then used to determine the crack density, crack velocity and crack nucleation rate. These variables depend on the service conditions (frequency, R-Ratio and test duration) the sample has experienced over the course of the experiment. Such values provide the necessary information to evaluate the performance of the material under the given conditions, as well as provide information to be used for subsequent lifetime expectancy calculations.

CHAPTER 5
RESULTS

Low frequency fatigue tests were conducted in order to analyze different fatigue parameters to quantify the effect on the SCC of X-65 carbon steel. The effect of surface finish on SCC behavior is also analyzed by preparing the test sample in different methods (i.e. sandblasting, polishing and acid pickling). The effects of constant load/displacement on SCC behavior followed by low frequency cyclic stress parameters were analyzed. The effects of oxygen presence and test durations were also determined.

1. Surface Finish.

The surface finish effect is analyzed using various methods. Different preparations methods are conducted on the test sample. These methods include sandblasting at around 25psi, polishing up to 1000 grit, acid pickling using Clark's solution, or leaving the sample as is (mill scale). Another method used is by polishing half the sample and leaving the other half as mill scale. The mill scale is composed of de-carburized layers, which results in a very rough surface. Figure 3 illustrates the different crack lengths and their respective maximum crack length observed for each surface roughness.

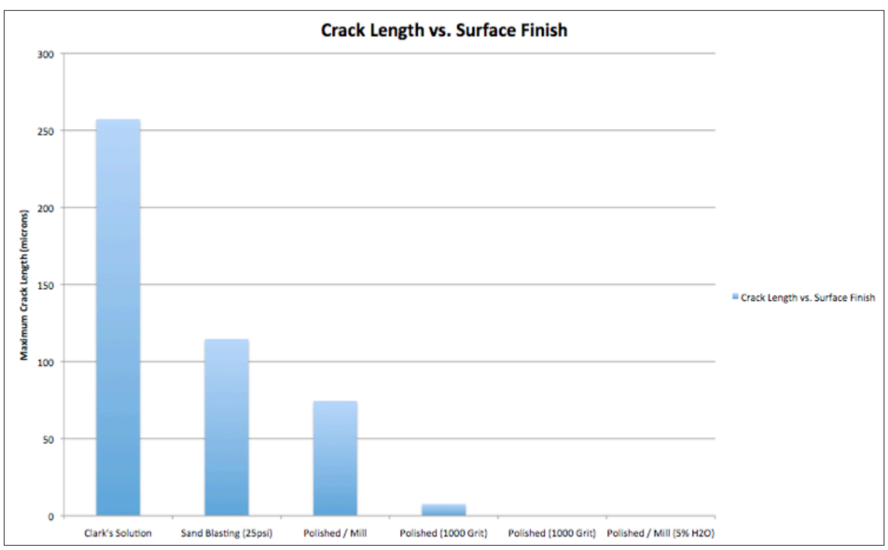

Figure 7. Crack Length vs. Surface Finish

As can be seen from the figure, the more polished the test sample is, the smaller the crack lengths observed are. Acid pickling provided the largest observable crack length. This is due to the fact that Clark's solution is an acid, which makes the metal's electrochemical potential drop further into the active region.

2. Constant Load/Displacement:

In this experiment, various loads (resulting in different displacements) are introduced to the sample and stabilized for a total exposure time of up to 360 hours. Table 1 illustrates the resultant data.

Table 2. Constant Load/Displacement Test Results

FPB SP #	Environment	Purged Gas	Max Load lbs	Displacement mm	Exposure Time hrs.	Remarks
1	SFGE-3	Air (cont.)	1400	0.25	360	No SCC
2	SFGE-3	Air (cont.)	4500	1.00	360	No SCC
3	SFGE-3	Air (cont.)	6500	2.50	360	No SCC
4	Pure FGE - S	Air (cont.)	6500	2.50	204	No SCC
5	Pure FGE - WOI	Air (cont.)	6500	2.50	360	No SCC

As can be observed, no SCC was detected with any of the constant load/displacement tests conducted, despite the presence of oxygen and chlorides in the system.

3. R-Ratio:

For this experiment, various R-ratios (0.03 and 0.75) are analyzed with all other parameters kept constant. The resultant data is illustrated in Figure 4 below.

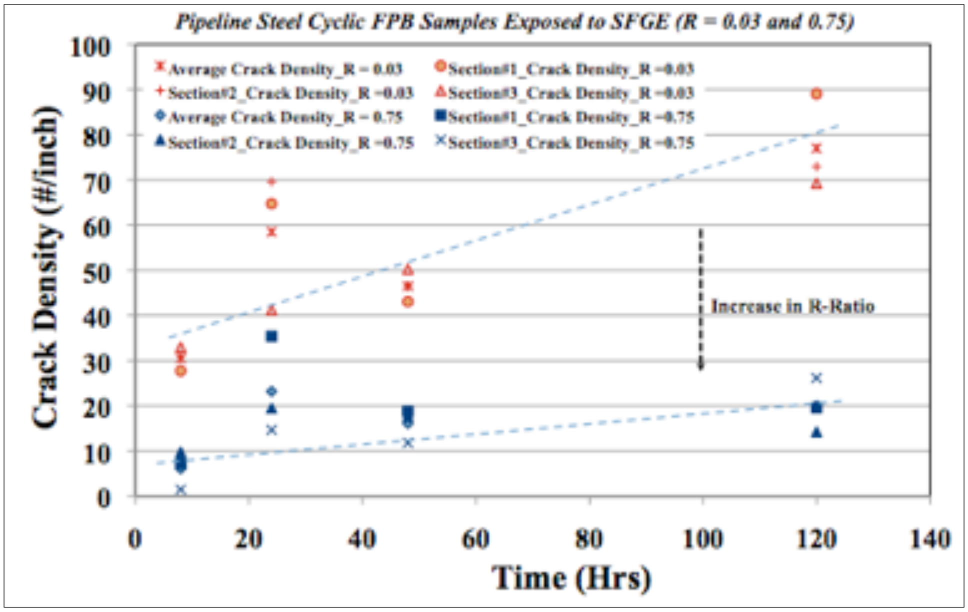

Figure 8A. R-Ratio Effect on Crack Density

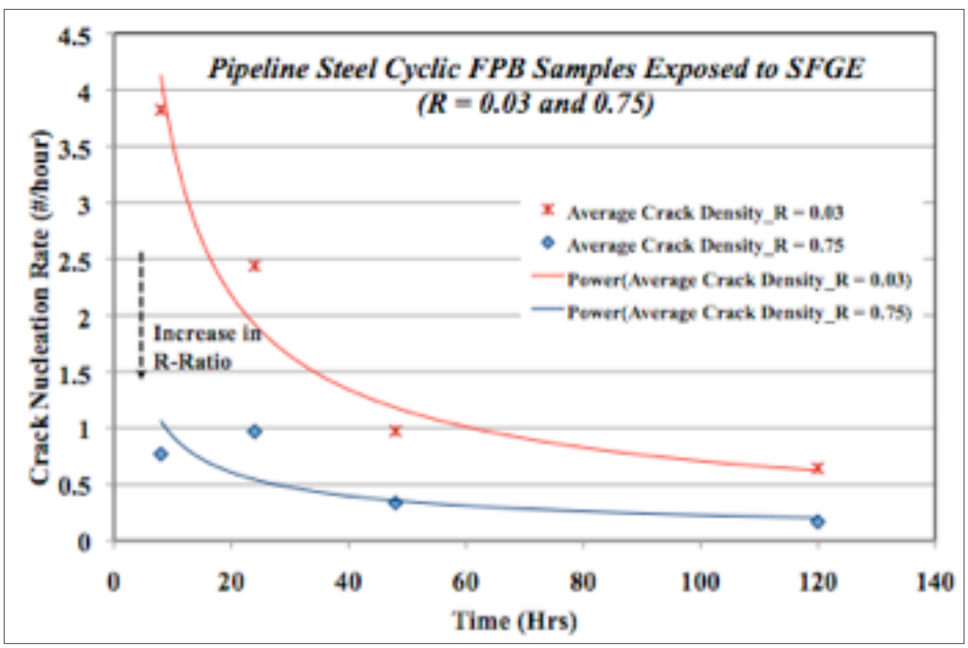

Figure 8B. R-Ratio Effect on Crack Nucleation Rate

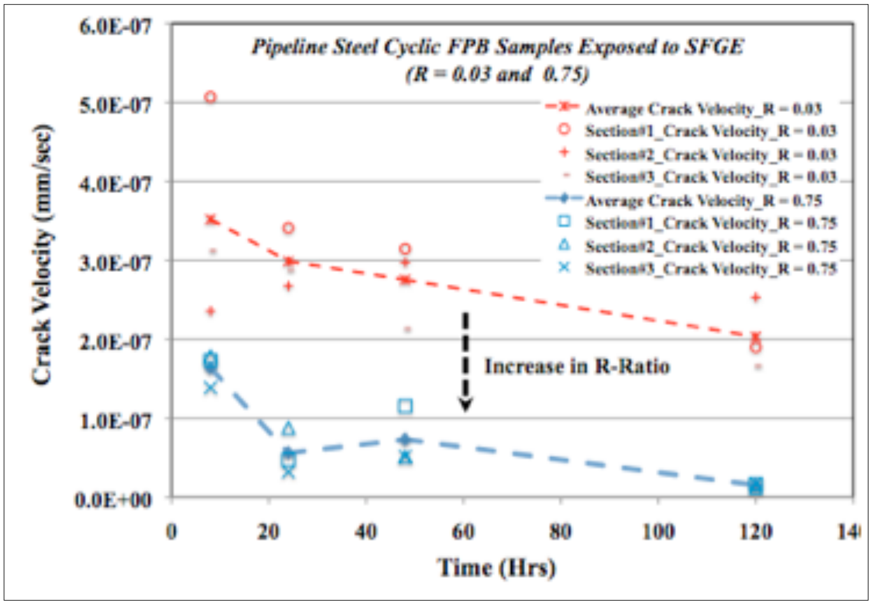

Figure 8C. R-Ratio Effect on Crack Velocity

As can be seen from the figures, the crack density (number of cracks per unit length), crack nucleation rate (how fast the crack nucleates) and crack velocity (how fast the crack propagates) all decrease with increasing R-ratios (i.e. less hoop stress fluctuations). This indicates that a smaller load range (difference between maximum and minimum loads applied) results in less severe SCC behavior. This behavior adequately fits the constant load/displacement tests, which had an R-ratio of 0. Hence, it can be conjectured that as the R-ratio (load variation) decreases, SCC behavior becomes more benign and less detrimental (vice versa).

4. Cycle Frequency:

For this experiment, various cycle frequencies are analyzed. The cyclic loading period ranged from $1.15x10^{-5}$ Hz to approximately $1x10^{-2}$ Hz. The test duration for all tests illustrated below is 10 days, with a load range of 200lbf to 6,500lbf. The resultant maximum crack length and crack velocity values for each test are illustrated in Figures 9A and 9B below.

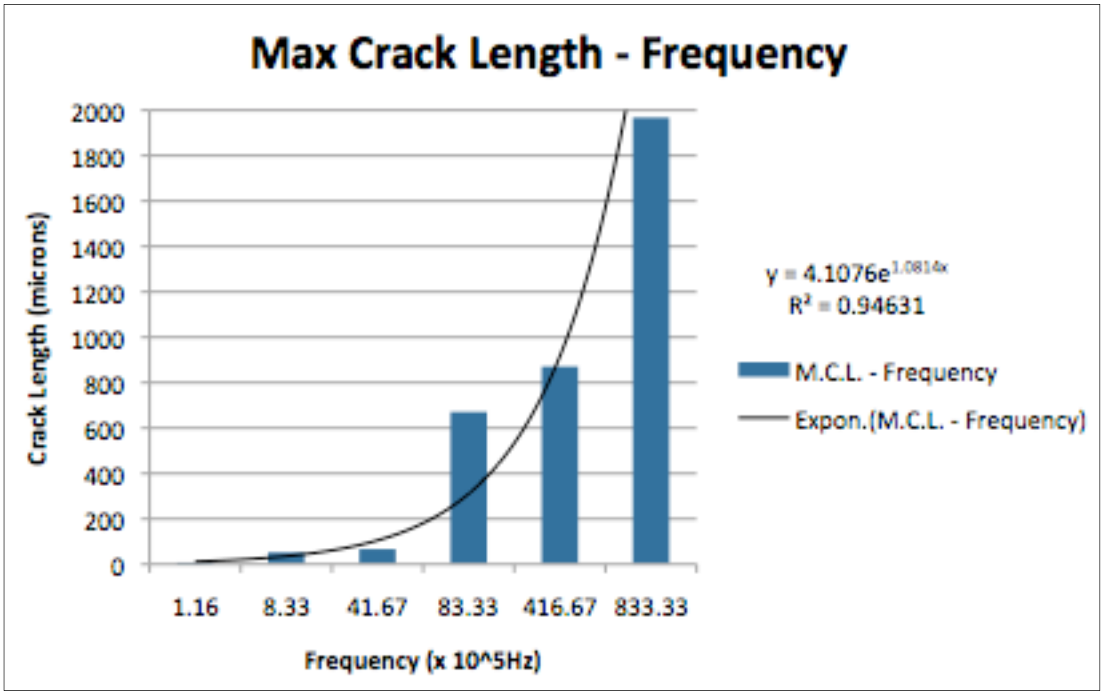

Figure 9A. Cyclic Frequency Effect on Maximum Crack Length

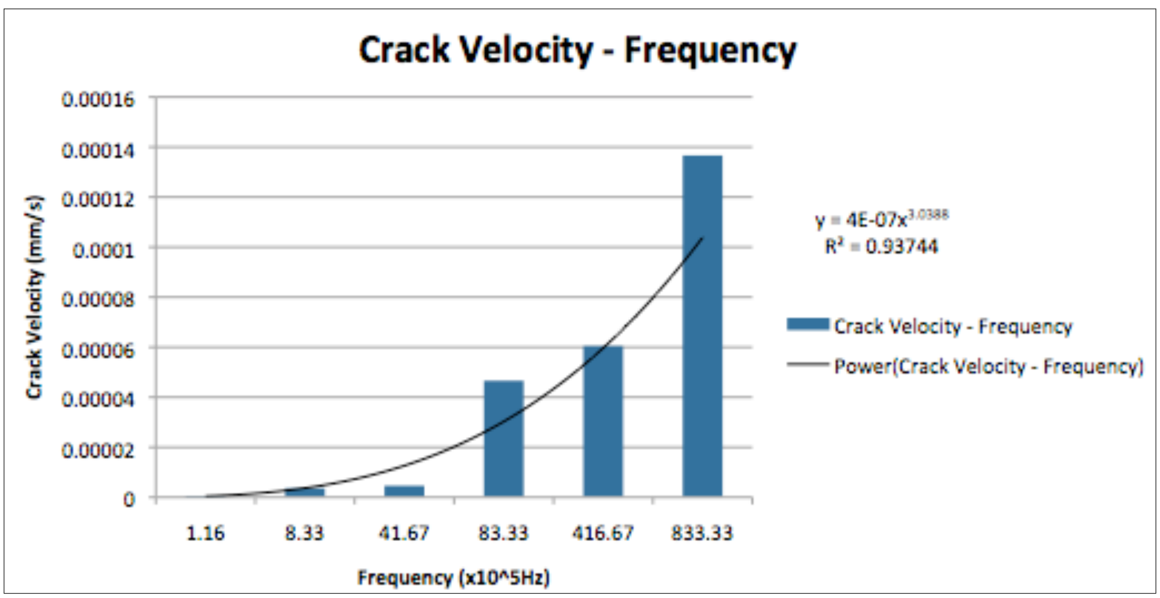

Figure 9B. Cyclic Frequency Effect on Crack Velocity

It can be observed that the maximum crack length and crack velocity increase in an exponential fashion with increasing cyclic frequency, with crack lengths reaching around 1900 microns, or 1.9mm (therefore macroscopically visible). Figures 9C and 9D illustrate the resultant crack density and nucleation rates of the tests conducted.

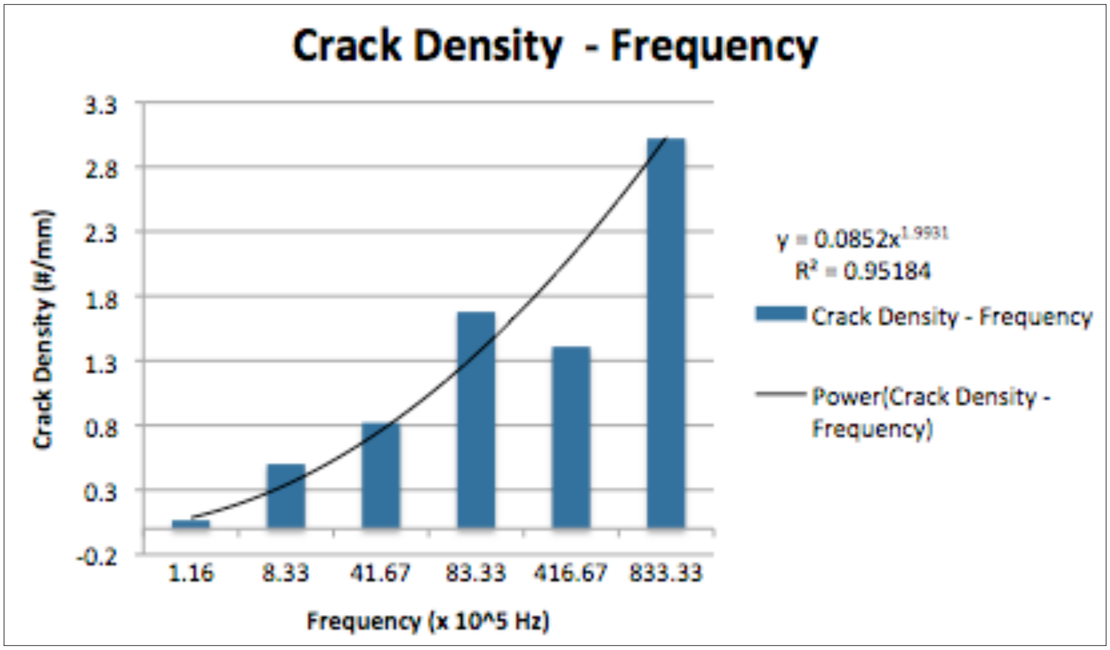

Figure 9C. Cyclic Frequency Effect on Crack Density

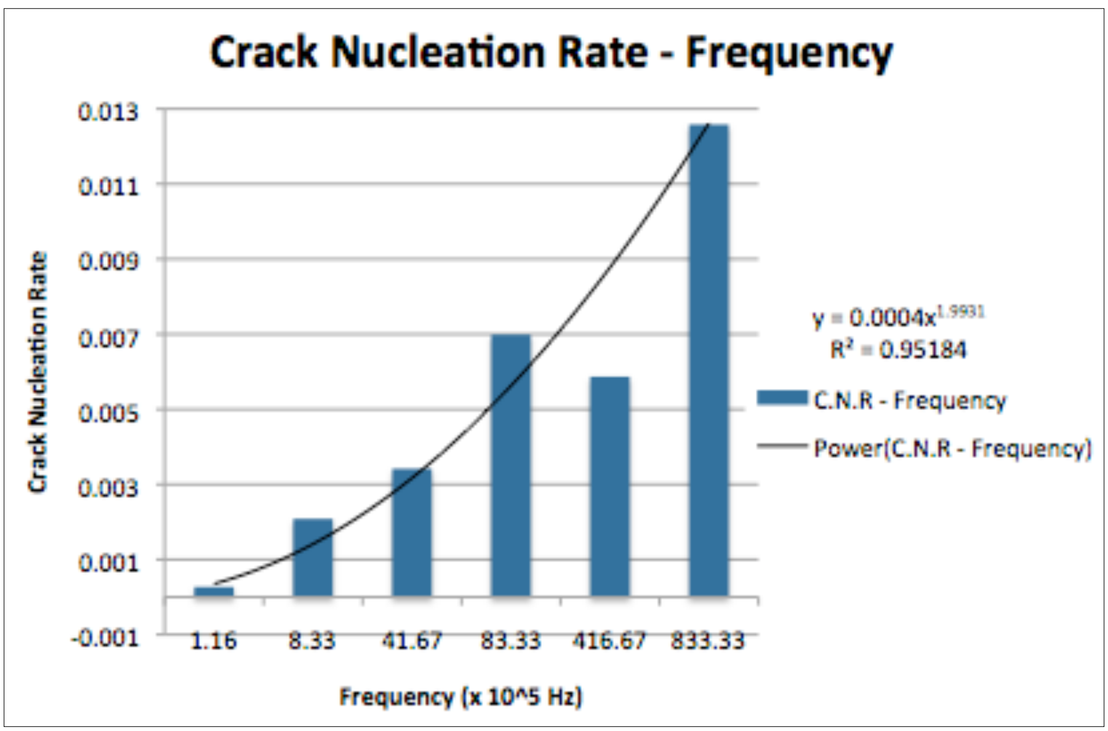

Figure 9D. Cyclic Frequency Effect on Crack Nucleation Rate

As can be seen from the figures, the crack density and nucleation rate increase with increasing cyclic frequency until 4min/cycle, which experiences a small drop. However, at higher frequencies, the density and nucleation rates increase dramatically. This potentially marks the onset of SCC crack propagation behavior.

5. Aeration Effects:

For this experiment, the effect of oxygen presence is analyzed. Two tests are conducted with a load range of 200lbf to 6500lbf with a cyclic period of 4 min/cycle for a test duration of 10 days. One test includes oxygen flow using the oxygen purge mechanism, while the other has no oxygen supplied to the solution. Figure 10 illustrates the results obtained from this experiment.

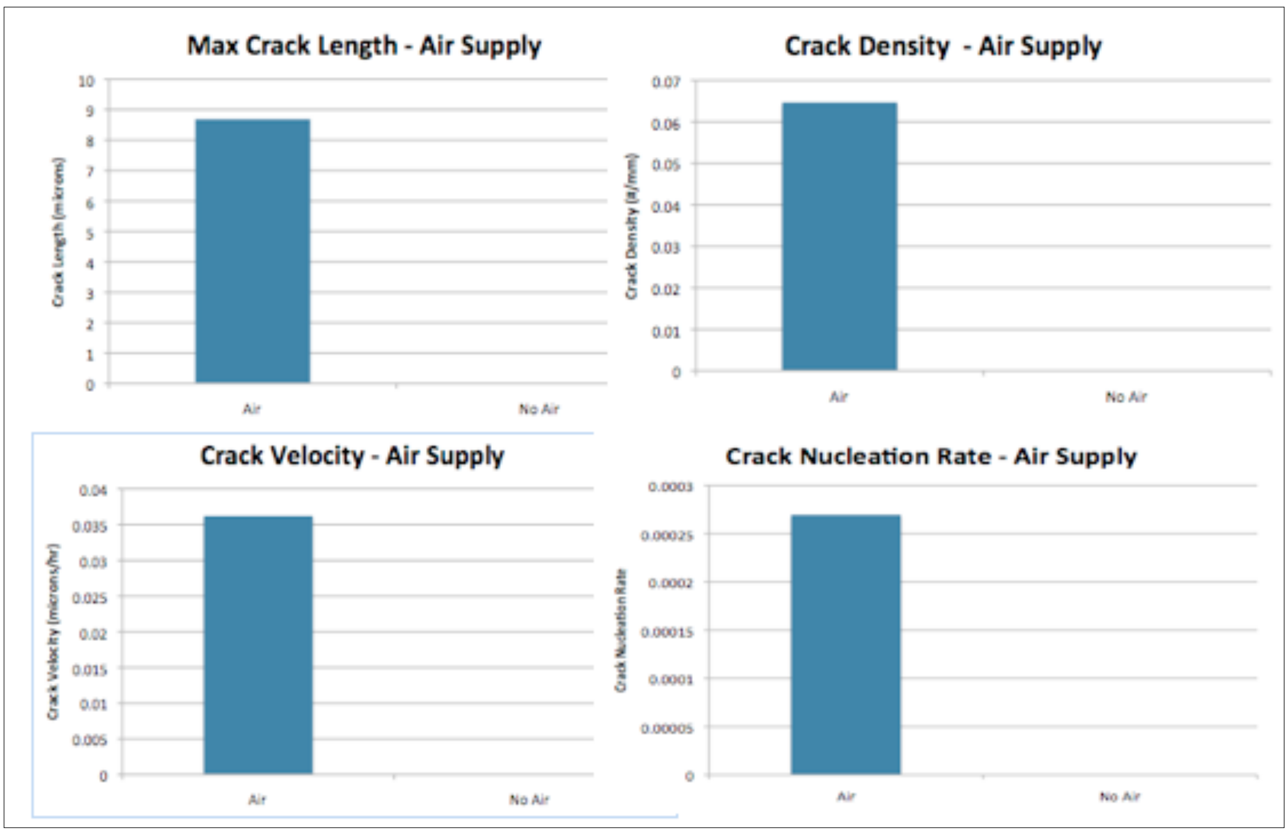

Figure 10. Aeration Effects on SCC Behavior

As can be seen from the figure, the presence of oxygen results in crack initiation, with all values presented above being nonzero. However, when oxygen is not supplied, no cracking has been observed. This further supports previous research conducted by X.

Lou *et. al* and Sridhar *et. al*. Hence, the presence of oxygen as an oxidizing agent is essential for the carbon steel to reach the active region, therefore resulting in corrosive mechanisms to initiate and cracking to result.

6. Test Duration Effects:

For this experiment, the effect of test duration is analyzed. Two tests are conducted with a load range of 200lbf to 6,500lbf with a cyclic period of 4 min/cycle. One test was conducted for approximately 5 days, while the other was conducted for 10 days. Figure 11 illustrates the results obtained from this experiment.

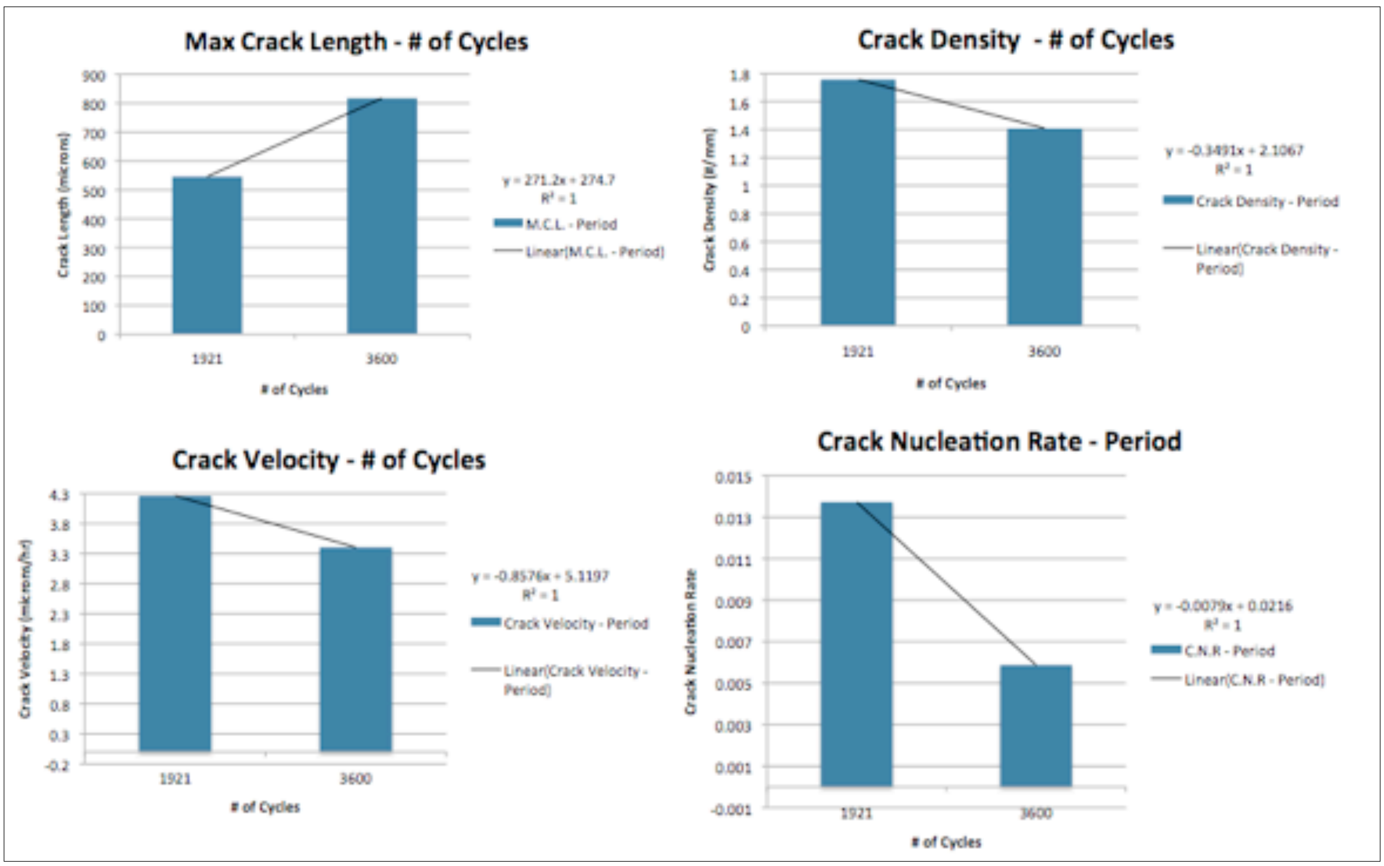

Figure 11. Test Duration Effects on SCC Behavior

As can be seen from the figure, longer test duration results in a larger maximum crack length. However, it also results in a reduced crack density, velocity and nucleation rate. This is most probably due to the fact that crack shielding occurs. This is a phenomenon that occurs when the plastic zones of the cracks in a component start interfering with each other, therefore slowing down each other's growth mechanisms. Furthermore, cracks close enough together can and do tend to merge together to form larger, more severe cracks. Hence, it can be conjectured that the crack-shielding phenomenon may result in the illustrated behavior as shown.

CHAPTER 6
FUTURE WORK

Further work needs to be done to better analyze SCC effects and behavior in X-65 carbon steel. Longer test durations need to be analyzed to examine the effects of long-term effects of cyclic loading on crack behavior. It is also important to determine whether seemingly harmless loading conditions may in fact not fully prevent cracking from initiating with longer exposure durations. Various load ranges need to also be examined, especially at below yield stress levels, in order to analyze how SCC can initiate and behave when full plasticity is not reached. Such experiments will provide a better perspective of the effect of time on crack behavior and propagation within the system at hand, and will thus result in a coherent, comprehensive study of SCC behavior in X-65 carbon steel pipelines.

CHAPTER 7
DISCUSSION / CONCLUSIONS

In this research project, various aspects are analyzed to examine SCC behavior of fuel-grade ethanol pipelines made of X-65 carbon steel. From the data presented above, the following conclusions are drawn:

- No SCC is experienced in constant load/displacement tests below or above yield stress levels, which indicates that cyclic loading is required for SCC to occur.
- A smoother surface induces significantly less SCC than a rougher surface, indicating that surface roughness plays a big role in crack initiation and propagation.
- Crack density, nucleation rate and velocity decrease as the R-ratio increases, which indicates that less severe stress fluctuations result in less SCC formation.
- Crack density, nucleation rate, crack velocity and maximum crack length increase with increasing frequency, with a possible threshold leading towards crack propagation mechanisms.
- Oxygen supply is essential for SCC to occur. In other words, the absence of an oxidizing agent will result in no SCC to be observed.
- A longer test duration results in a larger crack length but lower crack density, velocity and nucleation rate due to possible crack shielding and merging, with plastic zone interference between neighboring and adjacent cracks.

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